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Microwave dielectric characterization of hay during pyrolysis

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ABSTRACT

In this study, the microwave dielectric properties of hay were investigated at two frequencies (915 and 2450 MHz) from room temperature to ~700 °C in an inert environment. These properties were scrutinized in three distinct stages; namely drying (from room temperature to ~200 °C), pyrolysis (from ~200 °C to ~450 °C), and char region (from ~450 °C to ~700 °C). The dielectric properties were found to decrease during drying and pyrolysis stages, while increased significantly in the char region. The penetration depth of the microwave in hay is presented against the increasing temperature. Overall, the microwave absorption capability of the biomass material improved significantly after pyrolysis process, i.e. in the char region. Hay in its original form can be considered as a low loss dielectric material. However, its char product showed a good microwave absorbing capability. The experimental data were fitted using regression fit and based on this the dielectric properties model related to the temperature was developed. The result from this study can be useful to design and develop microwave system for hay.

1. Introduction

Hay is a mixture of grasses (ryegrass, timothy, etc.) and legumes (alfalfa, clovers, etc.) and other species depending on the region it is cultivated. Timothy which is considered as one of the main parts of hay is a Eurasian grass which was cultivated first in the United States in 1711 (Hoover et al., 1948), and then spread to Canada in 1747 (Smoliak et al., 1981). It can be found throughout U.S. and Canada except Prince Edward Island and Labrador (Bliss and Wein, 1972; Stubbendieck et al., 1986). It grows under a wide variety of range and soil moisture conditions (Plummer, 1955). It is basically the leaf and seed material in the hay that determines its quality. It is abundantly available and can be expected as a potential raw material for bio-products. For instance, very recently, hay was hydrolyzed to produce fermentable sugars (Orozco et al., 2013).

Pyrolysis is one of the thermochemical methods to complete destruction of organic materials into products of different form; solid, liquid, and gas. The quality of the product depends on the feedstock characteristics and process conditions (Fernández and Menéndez, 2011; Jahirul et al., 2012b; Masek et al., 2013; Mythili et al., 2013). Further, the pyrolysis products can be upgraded into chemicals and fuels (Bridgwater, 1996; Czernik and Bridgwater, 2004). Hence, pyrolysis process has received considerable attention during the past few decades (Bridgwater, 2012).

Several technologies including fixed bed, fluidized bed, transported bed, augur or screw, ablative, rotating cone, chain gates, etc. (Bridgwater, 2012) have been developed to carry out the pyrolysis process. However, each technology has its own advantages and disadvantages. Typically, the heat is transferred to the materials via conduction or convection in conventional heating methods. However, these methods suffer from high resistance to heat transfer which is one of the major drawbacks of conventional heating. Several researchers as reviewed by Motasemi and Afzal (2013) have conducted research on microwave assisted pyrolysis in which the heat can be directly generated within the material (Kappe et al., 2012).

Microwave pyrolysis has attracted a considerable attention during the past decade because of several advantages such as rapid and volumetric heating which increases the heating efficiencies (Appleton et al., 2005; Arenillas et al., 2011; Jahirul et al., 2012a), save time and energy, and improved product quality as compared to other methods (Abubakar et al., 2013). Various types of biomass were pyrolyzed under microwave irradiation; namely switchgrass (Zhou et al., 2013), wheat straws (Zhao et al., 2011, 2013), rice straws (Du et al., 2010; Huang et al., 2010), corn stover (Huang et al., 2013; Wang et al., 2012), algae (Wan et al., 2010), coffee hulls (Domínguez et al., 2007; Menéndez et al., 2007), oil palm shell (Salema and Ani, 2012), wood (Chen et al., 2008; Miura et al., 2000, 2004; Wang et al., 2008).

Before processing any material under microwave, it is essential to understand the fundamental dielectric properties of the material such as dielectric constant, loss factor, tangent loss, and microwave

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Table 1
Proximate and ultimate analysis of hay.

Proximate analysis (wt.%, dry basis)	
Ash	4.06
Volatile	79.55
Fixed carbon	13.57
Moisture content	2.82
Ultimate analysis (wt.%, dry, ash free)	
Carbon	45.46
Nitrogen	0.18
Hydrogen	5.95
Sulphur	0.04
Oxygen	48.37

absorption capacity. Usually these properties vary with temperature and microwave frequency (Motasemi et al., 2014; Peng et al., 2012; Salema et al., 2013; Yu et al., 2001). Therefore, it is important to investigate such changes in dielectric properties during actual pyrolysis process (Peng et al., 2012). Although numerous research work has been carried out on microwave pyrolysis of biomass, but there is a clear lack of dielectric properties for agricultural biomass in the literature. In particular, there was no published data on the microwave dielectric properties of hay. Studies of dielectric properties for other materials such as, peanut hull pellets (Paz et al., 2010a), woody biomass (Olmi et al., 2000; Ramasamy and Moghtaderi, 2010), hardwood (Sahin and Ay, 2004), Aleppo pine, Holm oak and Thuja burl woods (El Alami et al., 2012), softwoods (black spruce, Balsam fir, and Tamarack) (Afzal et al., 2003), oil palm biomass (Salema et al., 2013), empty fruit bunch (Omar et al., 2011), and wood and wood-based products (Torgovnikov, 1993) could be found.

Based on the available literature and to our knowledge this study is of first kind to reveal the microwave dielectric properties of hay during pyrolysis. The dielectric properties consisted of dielectric constant, loss factor, tangent loss and microwave penetration depth. The dielectric properties were determined from room temperature to ~700 °C and at two known frequencies (915 MHz and 2450 MHz). Lastly, the microwave absorption behaviour during pyrolysis was fitted to a numerical function in order to develop dielectric properties model. The knowledge of dielectric properties and related data will be helpful to evaluate the penetration depth of microwave in the material and design the large scale microwave heating system (Meredith and Engineers, 1998). Thus, the dielectric properties of hay were measured in order to investigate: (i) the heating characteristics of material, (ii) the changes in microwave absorption characteristics during processing, and (iii) the best choice of frequency for heating/processing.

2. Material and methods

2.1. Material

Hay was used as the raw material in the present study. The proximate and ultimate analyses of hay are presented in Table 1. The sample was ground in a Wiley mill to particle size of less than 2 mm. The hay powder was dried at 105 °C for 15 h in a conventional oven, and then were uniaxially pressed (a die lined with tungsten carbide) at ~135 MPa. The dielectric properties were investigated on three hay pellets with similar dimensions stacked on top of each other to form one test sample. The physical properties of the hay pellets were; diameter (3.75 ± 0.050 mm), length (11.95 ± 0.050 mm), mass (0.10 ± 0.002 g), and initial density (0.76 ± 0.050 g/cc). The purpose of pressing samples into pellet form was to eliminate the influence of air during measurement of dielectric property. Moreover, smaller sized pellets increased the uniformity of microwaves in the sample as well as the accuracy and reliability of the data.

2.2. Measurement of dielectric properties

The dielectric response of a substance is commonly presented as permittivity:

$$\varepsilon = \varepsilon_0 \varepsilon_r = \varepsilon_0 \varepsilon'_r - j\varepsilon''_r) \quad (1)$$

where, ε_0 is permittivity of free space (8.854×10^{-12} F/m), ε_r is complex relative permittivity, and j is imaginary unit ($j^2 = -1$). The values of permittivity for most materials are quite small. Therefore, complex relative permittivity is usually used instead of permittivity to describe the dielectric response. The complex relative permittivity is comprised of two components: (ε'_r) the real part (relative dielectric constant) and (ε''_r) imaginary part (relative loss factor) (Torgovnikov, 1993).

The dielectric properties of the hay (ε'_r and ε'') were measured using cavity perturbation technique. In this method the real time dielectric properties data can be acquired during pyrolysis at different temperature and frequencies. The detailed descriptions of the measurement technique and the apparatus can be found in previous articles (Peng et al., 2012). The major components of the measurement system consisted of resistive heating furnace and a cylindrical TM_0n_0 resonant mode cavity (ø 580 mm × 50 mm). In order to measure the permittivity at specific temperature, a hot sample and its holder are rapidly removed from a conventional furnace and inserted into a high electric field region of a thick-walled, well-cooled cavity. The resonant frequency and loaded Q of the cavity are measured by a Hewlett-Packard 8753 network analyzer and stored for off-line analysis which includes subtraction of hot empty sample holder effects. The sample and holder are either left in the cavity for further measurements at lower temperatures as the sample cools, or are quickly returned to the furnace for further processing. In the latter case, the sample can be out of the furnace for as little as 2 s for a measurement at a single frequency. In the cavity perturbation method, the differences in the microwave cavity response between a cavity with an empty sample-holder and the same cavity with a sample-holder plus the sample were measured. These differences were then used to calculate the permittivity (Pickles et al., 2005). In order to further minimize the error, the cavity perturbation method was first calibrated with high-purity sapphire solid sample and the second calibration point was measured with Teflon to check low dielectric constant values. The error of this calibration technique for the present sample length to diameter ratio (>3.5) was shown to be less than ±3%.

The samples were heated to the desired temperatures with a ramp rate of 5 °C/min, starting at ~30 °C and progressing in ~25 °C steps up to 150 °C, then 50 °C steps to ~700 °C. The pyrolysis reaction was done with the sample pellets mounted inside a 4 mm ID quartz tube with a 0.01 L/min flow of nitrogen (N_2) in order to avoid the oxidation and combustion.

The literature indicates that if the loss factor (ε''_r) is caused by free electron conductivity, there is a relationship between the dc resistance and the microwave loss factor (Ritz and Dressel, 2008). Making this assumption for the final "char" state of the sample, the final state loss factor at room temperature was estimated by measuring the final dc resistance of the final pellets. This relationship is expressed as follow (f is the microwave frequency, R is the resistance measured in the experiment, D is the diameter of the hay pellet, and L is the length of the hay pellet):

$$\varepsilon''_r = \frac{1}{2\pi f \varepsilon_0 R((\pi/4)(D^2/L))} \quad (2)$$

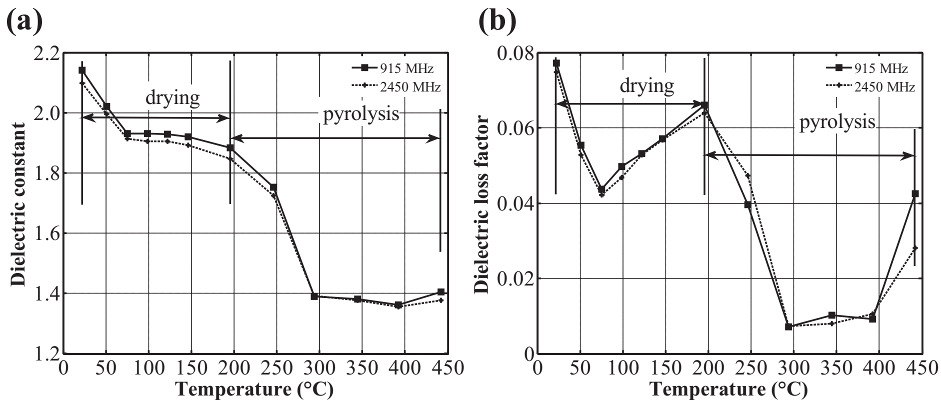

Fig. 1. Dielectric properties of hay vs. temperature under nitrogen environment at 915 MHz and 2450 MHz with initial density of 0.76 ± 0.05 g/cc; (a) relative dielectric constant and (b) relative loss factor.

The final dc resistance value was used to calculate a theoretical loss factor, which was then compared with the measured final loss factor as a test of the microwave loss mechanism.

2.3. Loss tangent

The loss tangent that represents the ability of the material to convert electromagnetic energy into heat at a specific temperature and frequency was calculated using the experimental data ($\tan\delta = \varepsilon''/\varepsilon'$). In any specific applicator system, the actual heating rate is also dependent on the oven geometry.

2.4. Penetration depth

The penetration depth (D_P) is a very important factor in the design and scale-up of a microwave heating system. It is defined as the depth in the material at which the power carried by a forward-travelling electromagnetic wave of the specified frequency falls to $1/e$ of the value it had just inside the surface. The penetration depth can be calculated by using the following equation (Meredith and Engineers, 1998) (λ_0 is the microwave wavelength in free space):

$$D_P = \frac{\lambda_0}{2\pi(2\varepsilon'_r)^{1/2}}\left\{\left[1+\left(\frac{\varepsilon''_r}{\varepsilon'_r}\right)^2\right]^{1/2}-1\right\}^{-1/2} \quad (3)$$

3. Results and discussion

The thermal decomposition of hay was divided into three distinct stages; namely, drying (from room temperature to 200 °C), pyrolysis (from 200 °C to 450 °C), and the char region (from 450 °C to 700 °C). According to the derivative thermogravimetric (DTG) analysis of hay, a significant loss of mass was found to occur in temperature range between 180 °C and 440 °C and that the thermal decomposition was completed at 550 °C. The dielectric properties measured in drying and pyrolysis stages are discussed in Section 3.1, whereas Section 3.2 reports the dielectric properties in char region.

3.1. Dielectric properties in drying and pyrolysis region

The dielectric properties of hay measured during drying and pyrolysis stages are shown in Fig. 1. The values of dielectric properties at 915 and 2450 MHz frequencies were almost identical. This proved that dielectric properties for hay were independent of frequencies. The relative dielectric constant and loss factor decreased sharply from room temperature to 75 °C. The dielectric constant almost remained constant from temperature 75 °C to ~200 °C, while loss factor increased sharply within this temperature. The exact reason for this unusual behaviour of dielectric properties in drying region is not known at present. However, release of free and lightly bound moisture content form the hay might show such effects (Zahn et al., 1986).

Dielectric properties (dielectric constant and loss factor) dropped from ~200 °C to ~300 °C in the pyrolysis region due to breaking of chemical bonds and release of the volatile matter (Naik et al., 2010). However, after 300 °C the dielectric properties almost remained constant, except the relative loss factor that showed sharp increases at 400 °C. The relative dielectric constant decreased by 12% and 27% during drying and pyrolysis, respectively. In drying stage, relative loss factor decreased firstly by 43% and then it increased by 34%, whereas in pyrolysis stage it decreased by 89%.

In the initial stage of pyrolysis, the dielectric properties are expected to be highly dependent on the moisture content of the material. Once the moisture gets evaporated from the sample, other components in the biomass do not show any variation in dielectric properties (Salema and Ani, 2011). This shows that hay could be considered as a poor absorber of microwave in the pyrolysis region. Robinson and co-worker (Robinson et al., 2009) observed similar trend of dielectric properties during pyrolysis of wood material. The stabilization of relative dielectric constant and loss factor after 300 °C during pyrolysis stage indicates the transformation of hay into carbonaceous char in this temperature range (Peng et al., 2011b).

The tangent loss for hay in drying and pyrolysis stage is presented in Fig. 2 and interestingly the trend was almost similar to that of dielectric loss factor. Basically, tangent loss indicates the ability of hay to convert electromagnetic energy into heat. Tangent

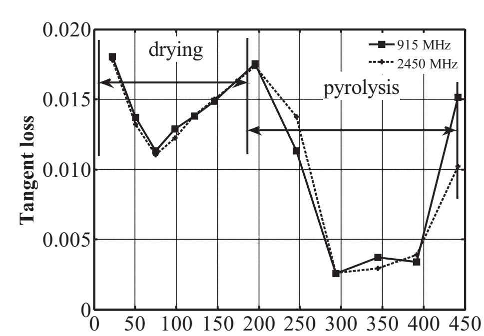

Fig. 2. Tangent loss for hay vs. temperature under nitrogen environment at 915 MHz and 2450 MHz.

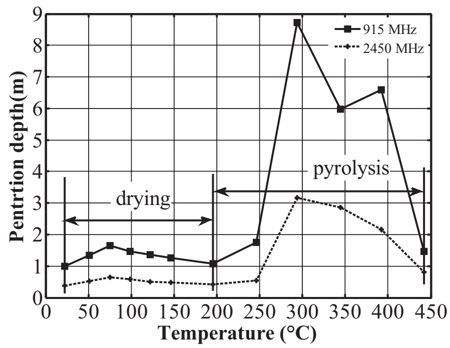

Fig. 3. Microwave penetration depth of hay at different temperature under nitrogen environment at 915 MHz and 2450 MHz.

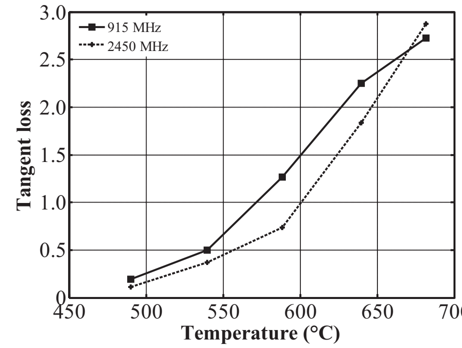

Fig. 5. Temperature dependences of tangent loss of hay-based biochar under nitrogen environment at 915 MHz and 2450 MHz.

loss decreased by 37% from room temperature to 75 °C as compared to 43% for loss factor.

Microwave penetration depth for hay during drying and pyrolysis processes is depicted in Fig. 3. The penetration depth remained relatively low and almost constant during drying phase. However, there was significant increase in the penetration depth during the pyrolysis reaction. Removal of moisture from the sample surface might have led sudden increase in the microwave penetration depth (Peng et al., 2012). Although the dielectric properties of hay were frequency independent, a notable difference was found in terms of penetration depth. The maximum penetration depth of 8.7 and 3.2 m was observed at 292 °C, for 915 and 2450 MHz frequencies, respectively. This difference could be due to the wavelength of the microwave at that particular frequency. Further, high penetration depth in the pyrolysis region indicates the transparency of the hay to the microwaves and in result the hay will not be able to absorb the microwave. Ultimately, a sudden drop in the penetration depth was observed at 400 °C.

3.2. Dielectric properties in char region

Dielectric properties of hay in char region are presented in Fig. 4. The result clearly indicates the high microwave absorption capability of the char. Unlike the drying and pyrolysis stage, the dielectric constant and loss factor in the char region showed an increasing trend in the temperature region of 450–700 °C. This shows the effect of loss of an insulating barrier due to change in the material characteristics (Peng et al., 2011b). It could be also due to thermal decomposition of hay, where the hydrocarbon gases and liquids are produced along with a solid char (Robinson et al., 2009). Process time and energy could be significantly compromised by elevating the temperature beyond 450 °C (Robinson et al., 2009). Therefore, researchers used char (Salema and Ani, 2011), activated carbon (Abubakar et al., 2013), and other carbonaceous materials (Menéndez et al., 2010) as a microwave absorber in order to increase the heat transfer and efficiency during microwave pyrolysis. Dielectric properties in the char region also depended on the applied frequency. Dielectric constant and loss factor were 1.6 times greater for 915 MHz than 2450 MHz. This could be attributed due to the inverse relationship between the dielectric constant and loss factor and microwave frequency (Paz et al., 2010b).

Similar results were reported by Peng et al. (2012) about the dielectric properties while pyrolyzing coal. In their study, the dielectric constant and loss factor was nearly constant up to 500 °C, but it increased significantly in the temperature range of 500–750 °C. In another study by the same authors (Peng et al., 2011a), they found that the complex permittivity increased with temperature but decreased with the frequency.

Fig. 5 depicts the tangent loss in the char region at 915 and 2450 MHz frequencies and from temperature 450 °C to 700 °C. The tangent loss increased significantly at both frequencies in this region. This indicates that char material can convert electromagnetic energy into heat.

The microwave penetration depth in hay based char is shown in Fig. 6. It decreased with increase in temperature. The penetration depth at 700 °C was lower than 2 mm which shows that microwave cannot penetrate the sample since its original size was about 3.5 mm. Hence, the char cannot be considered as transparent material; rather it is considered a high microwave absorption capability material. This significant decrease in penetration depth might be associated with the loss in volatile matter (Peng et al., 2012) which transforms the hay into carbonaceous material. Once the formation of carbon start to take place the microwave absorption capability of the material increase dramatically.

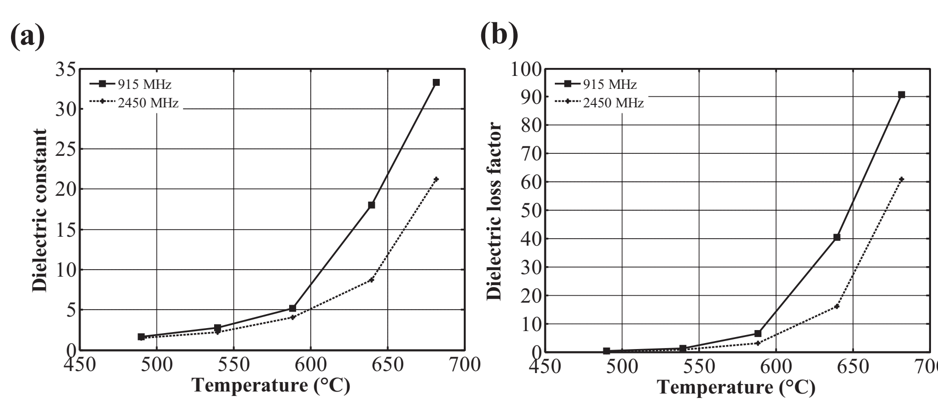

Fig. 4. Temperature dependences of dielectric properties of hay-based biochar under nitrogen environment at 915 MHz and 2450 MHz; (a) dielectric constant and (b) dielectric loss factor.

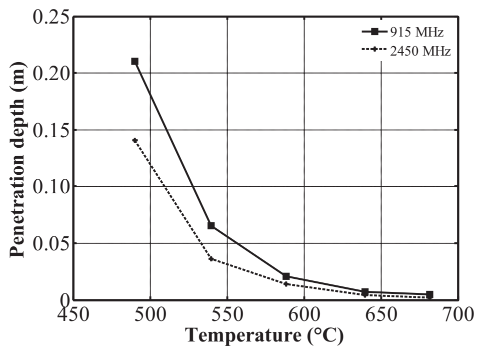

Fig. 6. Temperature dependences of microwave penetration depth of hay-based biochar under nitrogen environment at 915 MHz and 2450 MHz.

3.3. Dielectric properties model development

Fig. 7 shows the measured dielectric properties as a function of temperature at 915 and 2450 MHz frequency. The resulting best-fit generalized equation for dielectric properties was mostly obtained based on a sigmoidal function using regression model which is as follow:

$$y = \frac{A_1 - A_2}{1 + e^{(T - T_0)/dT}} + A_2 \tag{4}$$

where y is dielectric property (dielectric constant and loss factor); temperature, T, is the lone independent variable (°C) and A_i (i = 1, 2), T_0; and dT are the regression coefficients. In two cases, dielectric loss factor (drying) at 915 and 2450 MHz, Gauss function was used as the regression model as follow:

$$y = y_0 + \left(\frac{A}{W\sqrt{\pi/2}}\right) \times e^{[-2((T-T_c)/w)^2]} \tag{5}$$

where y is dielectric loss factor in this case; temperature, T is the lone independent variable (°C); and W, A, y_0, and T_c are the regression coefficients. The regression coefficients for different stage of

Table 2
Fit of relative dielectric constant model (Eq. (4)).

Stage	A_1	A_2	T_0	dT	R^2
915 MHz					
Drying	1.65	1.91	46.34	12.23	0.97
Pyrolysis	1.88	1.38	255.48	9.10	0.99
Char region	1.60	44.64	663.22	27.65	0.99
2450 MHz					
Drying	305.00	1.86	305.57	45.80	0.97
Pyrolysis	1.85	1.37	258.44	11.84	0.99
Char region	1.38	13,757.56	967.45	43.69	0.99

Table 3
Fit of relative dielectric loss factor model (Eq. (4)).

Stage	A_1	A_2	T_0	dT	R^2
915 MHz					
Pyrolysis	0.07	0.02	245.62	2.22	0.71
Char region	0.23	121.46	656.19	23.53	0.99
2450 MHz					
Pyrolysis	0.06	0.01	247.74	2.44	0.90
Char region	0.11	1029.74	765.46	30.31	0.99

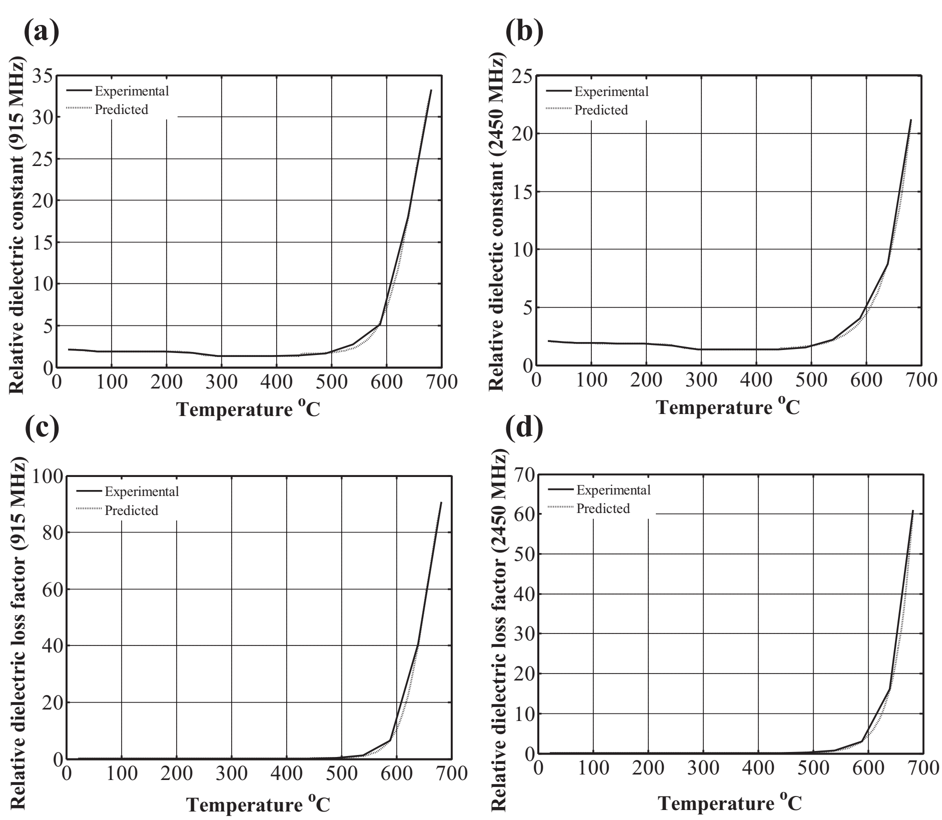

Fig. 7. Experimental and predicted values of dielectric constant of hay (a) and (b); loss factor (c) and (d)

Table 4
Fit of relative dielectric loss factor model (Eq. (5)).

Stage	y_0	x_c	W	A	R^2
915 MHz					
Drying	0.07	95.81	79.09	−2.63	0.74
2450 MHz					
Drying	0.07	91.42	68.95	−2.22	0.77

hay thermal degradation are provided in Tables 2 and 3 (for Eq. (4)), and Table 4 (for Eq. (5)).

The experimental data was fitted to Boltzmann and Gauss functions which had sigmoidal shapes. The equation describes the behaviour of relative dielectric constant and loss factor as a function of the temperature (T). The coefficients of determination (R^2) were between 0.71 and 0.99 which indicates how well the data points fits a line or curve (Steel and Torrie, 1960). The high values of R^2 (0.9 and above) denotes that the predicted values of dielectric properties derived using the derived equation were in close agreement with the experimental data as shown in Fig. 7.

4. Conclusions

The relative dielectric constant, loss factor, and tangent loss of the hay were measured at 915 and 2450 MHz frequencies from room temperature to ~700 °C and under nitrogen (N_2) environment. Overall, dielectric properties showed decreasing trend in drying and pyrolysis regions, but a sharp increase in the char region. This could be due to the transformation of biomass material into carbonaceous material, continuous change in weight and density, release of moisture and volatile matter. Hence, the dielectric properties tend to change with temperature and material characteristics. Low penetration depth during drying and char stage proves the microwave absorption capability of the hay and its char respectively. On the other hand, high penetration depth in the pyrolysis region could be due to transparency of microwave towards the material. The developed models represented the experimental behaviour of dielectric properties of hay very well. In conclusion, hay is considered as a low loss material, i.e. it is poor absorber of microwave, while its char exhibited strong microwave absorbing capability.

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